

(19)

Europäisches Patentamt  
European Patent Office  
Office européen des brevets



(11)

**EP 0 462 574 B2**

(12)

## NEW EUROPEAN PATENT SPECIFICATION

(45) Date of publication and mention  
of the opposition decision:  
19.12.2001 Bulletin 2001/51

(51) Int Cl.7: **D04H 1/54**

(45) Mention of the grant of the patent:  
10.05.1995 Bulletin 1995/19

(21) Application number: **91109987.7**

(22) Date of filing: **18.06.1991**

(54) **Nonwoven web and method of forming same**

Vliesstoff und Verfahren zu seiner Herstellung

Etoffe non-tissée et son procédé de réalisation

(84) Designated Contracting States:  
**BE DE ES FR GB IT NL SE**

(30) Priority: **18.06.1990 US 540070**

(43) Date of publication of application:  
**27.12.1991 Bulletin 1991/52**

(73) Proprietor: **KIMBERLY-CLARK WORLDWIDE,  
INC.  
Neenah, Wisconsin 54956 (US)**

(72) Inventors:

- Timmons, Terry Kramer  
Marietta, GA 30066 (US)
- Kobylivker, Peter Michalovich  
Marietta, GA 30068 (US)
- Woon, Lin-Sun  
Marietta, GA 30062 (US)

(74) Representative: Diehl, Hermann O. Th., Dr. et al  
Diehl, Glaeser, Hiltl & Partner Patentanwälte  
Postfach 34 01 15  
80098 München (DE)

(56) References cited:

EP-A- 0 316 195                      EP-A- 0 370 835  
US-A- 3 502 763                    US-A- 3 692 618  
US-A- 3 849 241                    US-A- 3 953 655  
US-A- 4 451 589

- Vollmert, "Grundriss der makromolekularen  
Chemie", Band III, E. Vollmert-Verlag, Karlsruhe,  
1988, p. 101
- Wadsworth, L.C. et al., "Effects of Melt Blown  
Web Structure on Filtration Efficiency and  
Porosity", Book of Papers, INDA-Tec,  
Philadelphia, Pennsylvania, USA, May 30-June 2  
1989, pp 585-600

- Galli, Simanazzi and del Duca, "New Frontiers In  
Polymer Blends: The Synthesis Alloys", Acta  
Polymerica 39 (1988) Nr. 1/2, Seiten 81-90
- E. Seiler, "Polypropylen/EPDM-Blends und  
Polypropylen-Impact-Copolymere, technische  
Werkstoffe im Fahrzeugbau und in der  
Elektrotechnik", 2. Symposium  
Thermoplastische Kunststoffe in technischen  
Wachstumsbranchen, SKZ Würzburg, 22-23.  
November 1988, Seiten 101-117
- Malkan, S.R.: "Process-Structure-Property  
Relationships in Different Molecular Weight  
Polypropylene Melt Blown Webs", Ph.D.  
Dissertation, The University of Tennessee,  
Knoxville, May 1990
- Cheng, C.Y. and Kwaiik, K.M.: "Polypropylene for  
Meltblown Applications", Meltblown  
Technology Today, Miller Freeman Publications  
, San Francisco, 1989, pp 61-63
- Gaides, F.C.: "Tailoring Polypropylene  
Polymers for Meltblown Applications",  
Meltblown Technology Today, Miller Freeman  
Publications, San Francisco, 1989, pp 61-63
- Malkan, S.R. et al.: "Process-Structure-Property  
Relationships in Melt Blowing of Different Melt  
Flow Rate Polypropylene Resins", in Turbak,  
A.F. and Vigo, T.L. Editors, Nonwovens: An  
Advanced Tutorial, Tappi Press, Atlanta, Usa,  
1989, pp 101-129
- Römpps Chemie-Lexikon, Franckh'sche  
Verlagshandlung, Stuttgart, 1974, pp 2200, 2201
- ASTM, Designation: D3593-80
- ASTM, Designation: D1238-95

**EP 0 462 574 B2**

## Description

[0001] This invention relates generally to a nonwoven web and a method of forming such a web.

[0002] Nonwoven fabric laminates are useful for a wide variety of applications. Such nonwoven fabric laminates are useful for wipers, towels, industrial garments, medical garments, medical drapes, and the like. Disposable fabric laminates have achieved especially widespread use in hospital operating rooms for drapes, gowns, towels, footcovers, sterile wraps, and the like. Such surgical fabric laminates are generally spun-bonded/melt-blown/spun-bonded (SMS) laminates consisting of nonwoven outer layers of spun-bonded polypropylene and an interior barrier layer of melt-blown polypropylene. Particularly, Kimberly-Clark Corporation, the assignee of the present invention, has for a number of years manufactured and sold SMS nonwoven surgical fabric laminates under the marks Spunguard® and Evolution®. Such SMS fabric laminates have outside spun-bonded layers which are durable and an internal melt-blown barrier layer which is porous but which inhibits the strikethrough of fluids from the outside of the fabric laminate to the inside. In order for such a surgical fabric to perform properly, it is necessary that the melt-blown barrier layer have a fiber size and a pore size distribution that assures breathability of the fabric while at the same time inhibiting strikethrough of fluids.

[0003] The current melt-blown web used in the manufacture of the Kimberly-Clark Evolution® medical fabric laminate has pore sizes distributed predominantly in the range from 10 to 15  $\mu\text{m}$  with the peak of the pore size distribution greater than 10  $\mu\text{m}$ . Such a melt-blown web has advantages as a barrier layer.

[0004] The article "Effects of Meltblown Web Structure on Filtration Efficiency and Porosity" (Wadsworth L.C. et al, Book of Papers, INDA-TEC, Philadelphia, Pennsylvania, USA, May 30 - June 2, 1989, pp. 585-600) relates to studies of polypropylene melt blown webs in terms of relationships among the processing conditions, structure and filtration efficiency. One of the resins used has a melt flow rate of 1400 and a polydispersity of 2.1.

[0005] It is an object of the present invention to further improve such nonwoven webs. This object is solved by the nonwoven web of independent claim 1 and the method of independent claim 5 and the nonwoven fabric laminate of independent claim 10. Further features and details of the invention are evident from the dependent claims, the description, examples and drawings.

[0006] The invention provides a nonwoven web having fine fibers and a small pore size distribution and a method for forming such a web. The method of the present invention uses a reactor granule resin having an initial broad molecular weight distribution which resin has been modified to narrow its molecular weight distribution and to increase its melt flow rate. Consequently the nonwoven web can be formed by melt-blowing at high throughputs. Such nonwoven webs are particularly useful as barrier layers for fabric laminates. The present invention allows a significant improvement in porosity and inhibition of strikethrough, and provides a melt-blown web having average fiber sizes of from 1 to 3  $\mu\text{m}$  and having a distribution of pore sizes so that the majority of pores are in the range of 7 to 12  $\mu\text{m}$  with the peak of the pore size distribution less than 10  $\mu\text{m}$ . More particularly, improved performance characteristics with respect to porosity and strikethrough can be achieved when the melt-blown web has pore sizes distributed predominantly in the range from 7 to 12  $\mu\text{m}$ , with a lesser amount of pores from 12 to 25  $\mu\text{m}$ , and with virtually no pores greater than 25  $\mu\text{m}$  as measure by the Coulter Porometer.

[0007] The present invention, therefore, provides a nonwoven web for use as a barrier layer in a fabric laminate which nonwoven web has an average fiber diameter of from 1 to 3  $\mu\text{m}$  and pore sizes distributed predominantly in the range from 7 to 12  $\mu\text{m}$ , with a lesser amount of pores from 12 to 25  $\mu\text{m}$ , with virtually no pores greater than 25  $\mu\text{m}$ , and with the peak of the pore size distribution less than 10  $\mu\text{m}$ .

[0008] The present invention also provides a nonwoven fabric laminate having a barrier layer of fine fibers and small pore size distribution such that the resulting fabric laminate has pore sizes distributed predominantly in the range from 5 to 10  $\mu\text{m}$ , with a lesser amount of pores from 10 to 15  $\mu\text{m}$ , with virtually no pores greater than 22  $\mu\text{m}$ , and with the peak of the pore size distribution shifted downward by up to 5  $\mu\text{m}$  from the peak peak of the melt-blown web alone.

[0009] The foregoing aspects are preferably obtained by forming a melt-blown web from a resin having a broad molecular weight distribution and having a high melt flow rate which resin is modified by the addition of a small amount of peroxide prior to processing to achieve an even higher melt flow rate (lower viscosity). During the melt-blowing process, the modified reactor granule polymer has an increased melt flow rate between 800 up to 5000 gms/10 min at 230°C.

[0010] Particularly, a polypropylene resin in the form of a reactor granule having a starting molecular weight distribution of 4.0 to 4.5 Mw/Mn and a melt flow rate of from 1000 to 3000 gms/10 min. at 230° C is combined with a small amount of peroxide, less than 500 ppm, to produce a modified polypropylene having a very high melt flow rate of up to 5000 gms/10 min. at 230° C and a narrower molecular weight distribution of 2.8 to 3.5 Mw/Mn.

[0011] Most preferably, the starting polypropylene resin for the melt-blown web of the present invention is a polypropylene reactor granule which resin has a molecular weight distribution between 4.0 and 4.5 Mw/Mn, has a melt flow rate of about 2000 gms/10 min. at 230°C, and is treated with about 500 ppm of peroxide to produce a modified resin having a melt flow rate greater than 3000 gms/10 min. at 230°C and a molecular weight distribution of from 2.8 to 3.5 Mw/Mn. The broader molecular weight distribution at the high melt flow rate helps minimize production of lint and

polymer droplets.

[0012] Other objects and advantages of the invention will become apparent upon reading the following detailed description and upon reference to the drawings.

[0013] Figure 1 is a schematic diagram of a forming machine which is used in making the nonwoven fabric laminate including the melt-blown barrier layer of the present invention;

[0014] Fig. 2 is a cross section view of the nonwoven fabric laminate of the present invention showing the layer configuration including the internal melt-blown barrier layer made in accordance with the present invention;

[0015] Fig 3 is a graph showing the pore size distribution for a melt-blown web made in accordance with the present invention (Sample 1), an SMS fabric laminate incorporating such a melt-blown web as a barrier layer (Sample 2), a conventional melt-blown web (Sample 3), and a conventional SMS fabric laminate (Sample 4).

[0016] Turning to Figure 1, there is shown schematically a forming machine 10 which is used to produce an SMS fabric laminate 12 having a melt-blown barrier layer 32 in accordance with the present invention. Particularly, the forming machine 10 consists of an endless foraminous forming belt 14 wrapped around rollers 16 and 18 so that the belt 14 is driven in the direction shown by the arrows. The forming machine 10 has three stations, spun-bond station 20, melt-blown station 22, and spun-bond station 24. It should be understood that more than three forming stations may be utilized to build up layers of higher basis weight. Alternatively, each of the laminate layers may be formed separately, rolled, and later converted to the SMS fabric laminate off-line. In addition the fabric laminate 12 could be formed of more than or less than three layers depending on the requirements for the particular end use for the fabric laminate 12.

[0017] The spun-bond stations 20 and 24 are conventional extruders with spinnerettes which form continuous filaments of a polymer and deposit those filaments onto the forming belt 14 in a random interlaced fashion. The spun-bond stations 20 and 24 may include one or more spinnerette heads depending on the speed of the process and the particular polymer being used. Forming spun-bonded material is conventional in the art, and the design of such a spun-bonded forming station is thought to be well within the ability of those of ordinary skill in the art. The nonwoven spun-bonded webs 28 and 36 are prepared in conventional fashion such as illustrated by the following patents: Dorschner *et al.* United States Patent No. 3,692,618; Kinney United States Patent Nos. 3,338,992 and 3,341,394; Levy United States Patent No. 3,502,538; Hartmann United States Patent Nos. 3,502,763 and 3,909,009; Dobo *et al.* United States Patent No. 3,542,615; Harmon Canadian Patent No. 803,714; and Appel *et al.* United States Patent No. 4,340,563. Other methods for forming a nonwoven web having continuous filaments of a polymer are contemplated for use with the present invention.

[0018] Spun-bonded materials prepared with continuous filaments generally have at least three common features. First, the polymer is continuously extruded through a spinnerette to form discrete filaments. Thereafter, the filaments are drawn either mechanically or pneumatically without breaking in order to molecularly orient the polymer filaments and achieve tenacity. Lastly, the continuous filaments are deposited in a substantially random manner onto a carrier belt to form a web. Particularly, the spun-bond station 20 produces spun-bond filaments 26 from a fiber forming polymer. The filaments are randomly laid on the belt 14 to form a spun-bonded external layer 28. The fiber forming polymer is described in greater detail below.

[0019] The melt-blown station 22 consists of a die 31 which is used to form microfibers 30. The throughput of the die 31 is specified in pounds of polymer melt per inch of die width per hour (PIH)\*. As the thermoplastic polymer exits the die 31, high pressure fluid, usually air, attenuates and spreads the polymer stream to form microfibers 30. The microfibers 30 are randomly deposited on top of the spun-bond layer 28 and form a melt-blown layer 32. The construction and operation of the melt-blown station 22 for forming microfibers 30 and melt-blown layer 32 is considered conventional, and the design and operation are well within the ability of those of ordinary skill in the art. Such skill is demonstrated by NRL Report 4364, "Manufacture of Super-Fine Organic Fibers", by V.A. Wendt, E.L. Boon, and C.D. Fluharty; NRL Report 5265, "An Improved Device for the Formation of Super-Fine Thermoplastic Fibers", by K.D. Lawrence, R.T. Lukas, and J.A. Young; and United States Patent No. 3,849,241, issued November 19, 1974, to Buntin *et al.* Other methods for forming a nonwoven web of microfibers are contemplated for use with the present invention.

[0020] The melt-blown station 22 produces fine fibers 30 from a fiber forming polymer which will be described in greater detail below. The fibers 30 are randomly deposited on top of spun-bond layer 28 to form a melt-blown internal layer 32. For an SMS fabric laminate, for example, the melt-blown barrier layer 32 has a basis weight of preferably about 0.35-0.50 oz./yd.<sup>2</sup>\*\*.

[0021] After the internal layer 32 has been deposited by the melt-blown station 22 onto layer 28, spun-bond station 24 produces spun-bond filaments 34 which are deposited in random orientation on top of the melt-blown layer 32 to produce external spun-bond layer 36. For an SMS medical fabric laminate, for example, the layers 28 and 36 each have a basis weight of preferably from about 0.30 oz./yd.<sup>2</sup> to about 1.2 oz./yd.<sup>2</sup>.

[0022] The resulting SMS fabric laminate web 12 (Fig. 2) is then fed through bonding rolls 38 and 40. The surface of the bonding rolls 38 and 40 are provided with a raised pattern such as spots or grids. The bonding rolls are heated

\* PIH = 1 pound = 0.453 kg; 1 inch = 2.54 cm

\*\* 1 oz/yd<sup>2</sup> = 33.91 g/m<sup>2</sup>

to the softening temperature of the polymer used to form the layers of the web 12. As the web 12 passes between the heated bonding rolls 38 and 40, the material is compressed and heated by the bonding rolls in accordance with the pattern on the rolls to create a pattern of discrete areas, such as 41 shown in Fig. 2, which areas are bonded from layer to layer and are bonded with respect to the particular filaments and/or fibers within each layer. Such discrete area or spot bonding is well known in the art and can be carried out as described by means of heated rolls or by means of ultrasonic heating of the web 12 to produced discrete area thermally bonded filaments, fibers, and layers. In accordance with conventional practice described in Brock et al., United States Patent No. 4,041,203, it is preferable for the fibers of the melt-blown layer in the fabric laminate to fuse within the bond areas while the filaments of the spun-bonded layers retain their integrity in order to achieve good strength characteristics.

[0023] In accordance with the present invention, we have found that the throughput (PIH) of the die head 22 may be increased while at the same time providing fine fibers by using a reactor granule form of the polymer rather than a pelletized form which polymer in reactor granular form has a molecular weight distribution of 4.0 to 4.5 Mw/Mn and a melt flow rate of 1000 to 3000 gms/10 min at 230 °C. During the melt-blowing process, the modified reactor granule polymer has an increased melt flow rate from 800 up to 5000 gms/10 min at 230°C. By modifying the starting polymer, the resulting polymer will have a lower extensional viscosity, thus taking less force to attenuate the fibers as they exit the die 31. Therefore, with the same air flow, the higher melt flow polymer will produce finer fibers at commercially acceptable throughputs. A commercially acceptable throughput is above 3 PIH. Lower throughputs, however, will further reduce the fiber and pore sizes of the melt-blown layer 32.

[0024] The resulting melt-blown web 32 with its fine fibers and resulting small pore size distribution has superior barrier properties when incorporated into a fabric laminate. Particularly, the unlaminated melt-blown web 32 has an average fiber size of from 1 to 3 µm and pore sizes distributed predominantly in the range from 7 to 12 µm, with a lesser amount of pores from 12 to 25 µm, with virtually no pores greater than 25 µm, and with the peak of the pore size distribution less than 10 µm.

[0025] When the melt-blown web 32 is incorporated into the SMS fabric laminate 12, the peak of the pore size distribution in the resulting SMS fabric laminate is shifted downward by up to 5 µm. The SMS fabric laminate 12 has pore sizes distributed predominantly in the range from 5 to 10 µm, with a lesser amount of pores from 10 to 15 µm, with virtually no pores greater than 22 µm, and with the peak of the pore size distribution shifted downward by up to 5 µm.

[0026] Figure 3 shows the pore size distribution for a melt-blown web made in accordance with the present invention (Sample 1), an SMS fabric laminate made using the melt-blown web of the present invention (Sample 2), a conventional melt-blown web (Sample 3), and an SMS fabric laminate such as Kimberly-Clark's Evolution® SMS medical fabric laminate made using the conventional melt-blown web (Sample 4). Particularly, the melt-blown web of the present invention and the SMS fabric laminate of the present invention were made in accordance with Example 1 below.

[0027] The present invention is carried out with polypropylene.

[0028] The method used to achieve the high melt flow polymer which is useful in producing a nonwoven web of fine fibers at commercial production speeds is to start with a reactor granule polypropylene resin having a molecular weight distribution between 4.0 and 4.5 Mw/Mn and a high melt flow rate of 1000 to 3000 gms/10 min. at 230°C. A small amount of peroxide is added to the starting resin to modify the molecular weight distribution to a range of 2.8 to 3.5 Mw/Mn and to increase the melt flow rate up to 5000 gms/10 min at 230°C.

#### Example 1

[0029] In order to illustrate the foregoing invention, a melt-blown web was formed on a conventional melt-blowing forming line using the modified polymer of the present invention. In addition, an SMS fabric laminate was formed using the inventive melt-blown web as an internal barrier layer. The SMS fabric laminate had spun bonded layers formed in conventional fashion of polypropylene. The SMS fabric laminate was preferably formed on-line by a multi-station forming machine as illustrated in Fig. 1. The melt-blown web and melt-blown barrier layer for the SMS fabric laminate were formed from reactor granules of polypropylene having a starting molecular weight distribution between 4.0 and 4.5 Mw/Mn and a melt flow rate of about 2000 gms/10 min. at 230°C. The starting polypropylene resin was treated with about 500 ppm of peroxide to produce a resin having a melt flow rate greater than 3000 gms/10 min. at 230°C and a molecular weight distribution of from 2.8 to 3.5 Mw/Mn. The broader molecular weight distribution at the high melt flow rate helps minimize production of lint and polymer droplets.

[0030] The melt-blown web, prepared in accordance with the foregoing, had a basis weight of 0.50 oz./yd.<sup>2</sup> and was designated as Sample 1. The SMS fabric laminate, having a melt-blown internal barrier layer made in accordance with the present invention, had spun-bonded layers with a basis weight of 0.55 oz./yd.<sup>2</sup>, and the melt-blown barrier layer had a basis weight of 0.50 oz./yd.<sup>2</sup>. The inventive SMS fabric laminate was designated as Sample 2.

[0031] In addition, a conventional melt-blown web and a conventional SMS fabric laminate (Kimberly-Clark's Evolution® fabric laminate) having the same basis weights as the inventive web and inventive SMS fabric laminate were prepared as controls. The control melt-blown web was designated Sample 3, and the control SMS fabric laminate was

designated Sample 4. The Samples 1 through 4 possess the characteristics set forth in Tables 1 and 2 below:

**Table 1**

**% Pore Size Distribution**

	<u>0-5<math>\mu</math>m</u>	<u>5-10<math>\mu</math>m</u>	<u>10-15<math>\mu</math>m</u>	<u>15-20<math>\mu</math>m</u>
Sample 1		50.7	45.8	2.9
Sample 2	1.8	55.4	40.3	1.9
Sample 3		10.5	67.7	21.4
Sample 4	1.2	20.0	61.6	11.6
	<u>20-25<math>\mu</math>m</u>	<u>25-30<math>\mu</math>m</u>	<u>Maximum pore Size <math>\mu</math>m</u>	
Sample 1	0.6	0		
Sample 2	0.4	0		22.0
Sample 3	0.5	0.1		
Sample 4	1.2	0.9		38.2

[0032] The pore size distribution set out in Table 1 was measured by the Coulter Porometer. The pore size distribution set out in Table 1 is shown graphically in Fig. 3. The plots shown in Fig. 3 show the finer pore size distribution for Samples 1 and 2 as compared to Samples 3 and 4 respectively. The pore size distribution for the inventive web and inventive SMS fabric laminate is narrower than the conventional melt-blown web and conventional SMS fabric laminate. It should be noted that the pore size distribution for the inventive SMS fabric laminate has the peak of its curve shifted downward by up to 5  $\mu$ m from the peak of the melt-blown web alone before lamination. Apparently the lamination process and the additional spunbonded layers cause the pore structure to close up thereby increasing the barrier properties of the resulting fabric laminate. The distribution of the pore sizes predominantly between 5 to 10  $\mu$ m represents a fabric laminate (Sample 2) that is finer in its construction than conventional fabric laminates (Sample 4) with the resulting improved barrier properties.

[0033] The improved barrier properties of the inventive fabric laminate (Sample 2) as compared to the conventional fabric laminate (Sample 4) is shown in Table 2 below.

**Table 2**  
**Barrier Properties**

**Blood Strikethrough**

<b>t = 0 min.</b>	<b>t = 1 min.</b>
<b><u>p = 1 psi</u></b>	<b><u>p = 1 psi</u></b>

<b>Sample 2</b>	<b>2.5%</b>	<b>12.4%</b>
<b>Sample 4</b>	<b>10.6%</b>	<b>14.5%</b>

**Bacteria Filtration Efficiency**

<b>Sample 2</b>	<b>95.4%</b>
<b>Sample 4</b>	<b>91.9%</b>

[0034] The blood strike through was measured by the following procedure. A 7 in\*\*\*, by 9 in. piece of each sample fabric was laid on top of a similar sized piece of blotter paper. The blotter paper was supported on a water filled bladder which was in turn supported on a jack. The jack was equipped with a gauge to determine the force exerted from which the pressure exerted by the bladder on the blotter paper was calculated. A 1.4 gm sample of bovine blood was placed on top of the fabric sample and covered with a piece of plastic film. A stationary plate was located above the plastic film. The water bladder was then jacked up until a pressure of 1 psi\*\*\*\* was attained on the bottom of the blotter paper. As soon as the pressure was achieved, that pressure was held for the desired time. Once the time had elapsed, the pressure was released, and the blotter paper was removed and weighed. Based on the difference in weight of the blotter paper before and after, the percentage strike through was determined.

[0035] The test results indicate that the SMS fabric laminate made in accordance with the present invention has superior strike through characteristics especially for short elapsed times. Short elapsed times represent the situations that are most often encountered in medical use where blood generally will not remain for long on the drape or gown before it can run off.

[0036] The filter properties were measured to determine the ability of the SMS fabric laminate to block the penetration of air born bacteria. The samples were tested in accordance with Mil. Spec. 36954-C 4.4.1.1.1 and 4.4.1.2.

[0037] The 3.5% increase in efficiency within the plus 90% range represents a significant improvement in filtration and the ability to preclude the passage of air born bacteria.

**Claims**

1. A nonwoven web of fine fibres formed from a reactor granule of a polypropylene resin having a molecular weight distribution between 4.0 and 4.5 Mw/Mn and a melt flow rate of 1000 - 3000 g/10 min at 230°C which has been modified by adding a small amount of peroxide to modify the molecular weight distribution to the range of 2.8 to 3.5 Mw/Mn and to increase the melt flow rate up to 5000 g/10 min at 230°C, wherein the modified polypropylene results from adding up to 500 ppm of peroxide to the reactor granules prior to forming the web.
2. The nonwoven web of claim 1, wherein the modified polypropylene has a melt flow rate of 3000 - 5000 g/10 min. at 230°C.
3. The nonwoven web of one of the preceding claims, wherein the web is formed at a polymer throughput of greater

\*\*\*1 inch = 2.54 cm  
\*\*\*\*1 psi = 0.069 bar

than 0.535 kg per cm of die width per hour (3PIH).

4. The nonwoven web of one of the preceding claims, wherein the web has an average fibre size from 1 to 3  $\mu\text{m}$  and pore sizes distributed predominantly in the range from 7 to 12  $\mu\text{m}$  with the peak of the pore size distribution less than 10  $\mu\text{m}$ .
5. A method of forming a nonwoven web having an average fiber size of from 1 to 3  $\mu\text{m}$  and pore sizes distributed predominantly in the range from 7 to 12  $\mu\text{m}$ , with the peak of the pore size distribution less than 10  $\mu\text{m}$ , comprising the steps of melt-blowing a reactor granule of a modified polypropylene having a molecular weight distribution between 2.8 and 3.5 Mw/Mn and a melt flow rate of from 800 g/10 min to 5000 g/10 min, wherein the polymer is throughput at a rate greater than 0.535 kg per cm of die width per hour (3PIH).
6. A method of forming a nonwoven web according to claim 5, having fine fibres and a small pore size distribution, wherein the modified polypropylene has a melt flow rate of greater than 3000 g/10 min. at 230°C.
7. The method of claim 6, wherein the modified polymer results from adding up to 500 ppm of peroxide to the reactor granules prior to forming the nonwoven web.
8. Use of the nonwoven web according to one of claims 1 to 4 or made according to one of claims 5 to 7 as a barrier layer in a nonwoven fabric laminate.
9. The use of claim 8 wherein the resulting fabric laminate has pore sizes distributed predominantly in the range from 5 to 10  $\mu\text{m}$  with a lesser amount of pores from 10-15  $\mu\text{m}$  and virtually no pores greater than 22  $\mu\text{m}$ , with the peak of the pore size distribution being preferably shifted downwardly by up to 5  $\mu\text{m}$  from the peak of the melt-blown web alone.
10. A nonwoven fabric laminate having at least two layers, one of said layers comprising a nonwoven web according to one of claims 1 to 4 or made according to one of claims 5 to 7.
11. The nonwoven fabric laminate of claim 10, whereby said web is a barrier layer in the nonwoven fabric laminate.
12. The nonwoven fabric laminate of claim 11, wherein the nonwoven fabric laminate is a nonwoven SMS fabric laminate, having an internal barrier layer.
13. Use of the nonwoven fabric laminate according to one of claims 10 or 12 in a sterilisation wrap or a surgical fabric.

#### Patentansprüche

1. Vliesbahn aus feinen Fasern, die aus einem Reaktorgranulat eines Polypropylenharzes, mit einer Molekulargewichtsverteilung Mw/Mn zwischen 4,0 und 4,5 und einer Schmelzflussrate von 1000-3000 g/10 min bei 230 °C, das durch Zusatz einer kleinen Menge Peroxid modifiziert wurde, um die Molekulargewichtsverteilung Mw/Mn auf den Bereich von 2,8 bis 3,5 zu ändern und die Schmelzflussrate auf bis zu 5000 g/10 min bei 230 °C zu erhöhen, gebildet sind, wobei das modifizierte Polypropylen durch Zusatz von bis zu 500 ppm Peroxid zu dem Reaktorgranulat vor dem Bilden der Bahn erhalten wird.
2. Vliesbahn nach Anspruch 1, wobei das modifizierte Polypropylen eine Schmelzflussrate von 3000-5000 g/10 min bei 230 °C aufweist.
3. Vliesbahn nach einem der vorangehenden Ansprüche, wobei die Bahn bei einem Polymerdurchsatz von mehr als 0,535 kg pro cm Düsenbreite pro Stunde (3PIH) gebildet wird.
4. Vliesbahn nach einem der vorangehenden Ansprüche, wobei die Bahn eine durchschnittliche Fasergröße von 1 bis 3  $\mu\text{m}$  hat und Porengrößen vorwiegend im Bereich von 7 bis 12  $\mu\text{m}$  verteilt sind, wobei der Spitzenwert der Porengrößenverteilung kleiner als 10  $\mu\text{m}$  ist.
5. Verfahren zur Bildung einer Vliesbahn mit einer durchschnittlichen Fasergröße von 1 bis 3  $\mu\text{m}$  und Porengrößen, die vorwiegend im Bereich von 7 bis 12  $\mu\text{m}$  verteilt sind, wobei der Spitzenwert der Porengrößenverteilung kleiner

als 10 µm ist, umfassend die Schritte des Schmelzblasens eines Reaktorgranulats aus einem modifizierten Polypropylen mit einer Molekulargewichtsverteilung Mw/Mn zwischen 2,8 und 3,5 und einer Schmelzflussrate von 800 g/10 min bis 5000 g/10 min, wobei das Polymer bei einer Rate von mehr als 0,535 kg pro cm Düsenbreite pro Stunde (3PIH) durchgesetzt wird.

- 5 6. Verfahren zur Bildung einer Vliesbahn nach Anspruch 5, mit feinen Fasern und einer kleinen Porengrößenverteilung, wobei das modifizierte Polypropylen eine Schmelzflussrate von mehr als 3000 g/10 min bei 230 °C aufweist.
- 10 7. Verfahren nach Anspruch 6, wobei das modifizierte Polymer durch Zusatz von bis zu 500 ppm Peroxid zu dem Reaktorgranulat vor dem Bilden der Bahn erhalten wird.
8. Verwendung der Vliesbahn nach einem der Ansprüche 1 bis 4, oder hergestellt nach einem der Ansprüche 5 bis 7, als Sperrschicht in einem Vliesstofflaminat.
- 15 9. Verwendung nach Anspruch 8, wobei das erhaltene Stofflaminat Porengrößen aufweist, die vorwiegend im Bereich von 5 bis 10 µm verteilt sind, mit einer geringeren Menge an Poren von 10-15 µm und im Wesentlichen keinen Poren größer 22 µm, wobei der Spitzenwert der Porengrößenverteilung vorzugsweise um bis zu 5 µm von dem Spitzenwert der schmelzgeblasenen Bahn alleine nach unten verschoben ist.
- 20 10. Vliesstofflaminat mit mindestens zwei Schichten, wobei eine der Schichten eine Vliesbahn nach einem der Ansprüche 1 bis 4 oder hergestellt nach einem der Ansprüche 5 bis 7 umfaßt.
11. Vliesstofflaminat nach Anspruch 10, wobei die Bahn eine Sperrschicht in dem Vliesstofflaminat ist.
- 25 12. Vliesstofflaminat nach Anspruch 11, wobei das Vliesstofflaminat ein SMS-Vliesstofflaminat mit einer inneren Sperrschicht ist.
13. Verwendung des Vliesstofflaminats nach einem der Ansprüche 10 bis 12 in einem Sterilisationstuch oder einem chirurgischen Stoff.

#### Revendications

- 35 1. Nappe non tissée constituée de fibres minces formées à partir de granules réactionnels d'une résine de polypropylène ayant une distribution de poids moléculaires comprise entre 4,0 et 4,5 Mp/Mn et un débit d'écoulement à l'état fondu de 1000 à 3000 g/10 min. à 230°C qui a été modifiée par addition d'une petite quantité de peroxyde pour modifier la distribution de poids moléculaires pour l'amener dans la gamme de 2,8 à 3,5 Mp/Mn et pour augmenter le débit d'écoulement à l'état fondu jusqu'à 5.000 g/10 min. à 230°C, le polypropylène modifié résultant de l'addition d'une quantité allant jusqu'à 500 ppm de peroxyde aux granules réactionnels, préalablement à la formation de la nappe.
- 40 2. Nappe non tissée selon la revendication 1, dans laquelle le polypropylène modifié a un débit d'écoulement à l'état fondu de 3000 à 5000 g/10 min. à 230°C.
- 45 3. Nappe non tissée selon l'une des revendications précédentes, dans laquelle la nappe est formée à un débit de polymère supérieur à 0,535 kg/cm de largeur de filière par heure (3 livres/pouce/heure).
- 50 4. Nappe non tissée selon l'une des revendications précédentes, dans laquelle la nappe a une taille moyenne de fibres qui va de 1 à 3 µm et des tailles de pores distribuées majoritairement dans la gamme de 7 à 12 µm, le pic de la distribution des tailles de pores étant inférieur à 10 µm.
- 55 5. Procédé de formation d'une nappe non tissée ayant une taille moyenne de fibres qui va de 1 à 3 µm et des tailles de pores distribuées majoritairement dans la gamme de 7 à 12 µm, le pic de la distribution des tailles de pores étant inférieur à 10 µm, comprenant les étapes de fusion-soufflage de granules réactionnels d'un polypropylène modifié ayant une distribution de poids moléculaires comprise entre 2,8 et 3,5 Mp/Mn et un débit d'écoulement à l'état fondu compris entre 800 g/10 min. et 5000 g/10 min., ledit polymère étant produit à un débit supérieur à 0,535 kg/cm de largeur de filière par heure (3 livres/pouce/heure).



## EP 0 462 574 B2

6. Procédé de formation d'une nappe non tissée selon la revendication 5, ayant des fibres minces et une faible distribution de tailles de pores, ledit polypropylène modifié ayant un débit d'écoulement à l'état fondu supérieur à 3.000 g/10 min. à 230°C.

5 7. Procédé selon la revendication 6, dans lequel le polymère modifié résulte de l'addition d'une quantité allant jusqu'à 500 ppm de peroxyde aux granules réactionnels, préalablement à la formation de la nappe non tissée.

8. Utilisation de la nappe non tissée selon l'une des revendications 1 à 4 ou fabriquée selon l'une des revendications 5 à 7 en tant que couche barrière dans stratifié d'étoffe non tissée.

10 9. Utilisation selon la revendication 8, dans laquelle le stratifié d'étoffe obtenu a des tailles de pores distribuées majoritairement dans la gamme de 5 à 10  $\mu\text{m}$ , avec une quantité moindre de pores d'une taille allant de 10 à 15  $\mu\text{m}$  et pratiquement pas de pores d'une taille supérieure à 22  $\mu\text{m}$ , le pic de la distribution des tailles de pores étant de préférence décalé vers le bas d'une distance allant jusqu'à 5  $\mu\text{m}$  par rapport au pic de la nappe obtenue par fusion-soufflage seule.

15 10. Stratifié d'étoffe non tissée formé d'au moins deux couches, dont l'une est constituée d'une nappe non tissée selon l'une des revendications 1 à 4 ou fabriquée selon l'une des revendications 5 à 7.

20 11. Stratifié d'étoffe non tissée selon la revendication 10, dans lequel ladite nappe est une couche barrière dans le stratifié d'étoffe non tissée.

12. Stratifié d'étoffe non tissée selon la revendication 11, dans lequel le stratifié d'étoffe non tissée est un stratifié d'étoffe SMS non tissée, contenant une couche barrière interne.

25 13. Utilisation d'un stratifié d'étoffe non tissée selon l'une des revendications 10 ou 12 dans une enveloppe de stérilisation ou une étoffe chirurgicale.

30

35

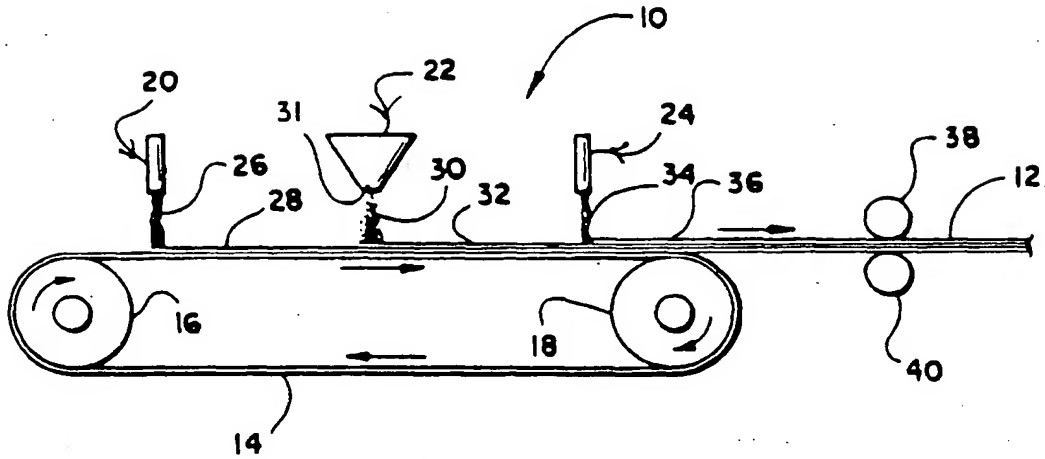
40

45

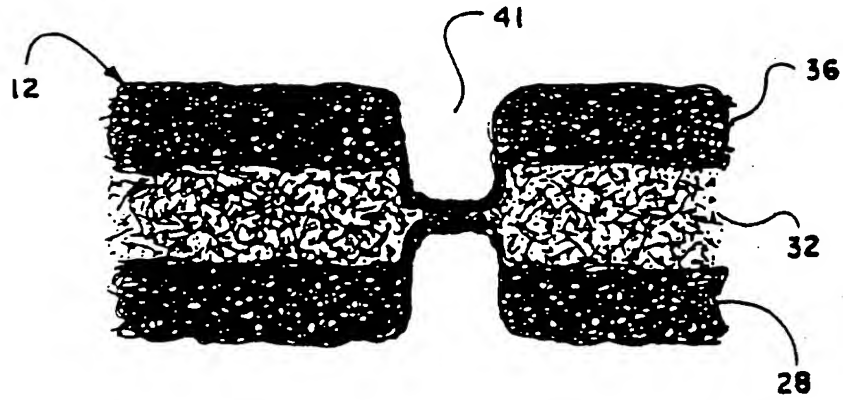
50

55

EP 0 462 574 B2



**Fig. 1**



**Fig. 2**

